



Fig.1 Relationship between average particle diameter of  $\text{Ca(OH)}_2$  and setting time



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF :  
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FOR: CEMENT SETTING ACCELERATOR :

DECLARATION UNDER 37 C.F.R. 1,132

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I, Hiroyoshi Kato, am one of the inventors of the present application.

I have conducted the following experiment to clarify that calcium hydroxide particles having an average particle diameter of 3  $\mu\text{m}$  or less are excellent in the effect of shortening the initial and final setting times of a cement composition as a cement setting accelerator.

<Experiment>

Runs 1 to 4

Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) particles having average particle diameters of 2.7  $\mu\text{m}$ , 2.5  $\mu\text{m}$ , 1.3  $\mu\text{m}$  and 0.5  $\mu\text{m}$  obtained by wet grinding using water as a dispersion medium were mixed in the form of a slurry with a cement and ion-exchanged water in an amount of 3.1 wt% based on the cement to obtain cement pastes so as to measure their setting times. A commercially available normal Portland cement was used as the cement.

Comparative Run 1

The setting time of a cement paste which was prepared under the same conditions as in Run 1 except that calcium hydroxide was not added was measured.

### Comparative Runs 2 to 6

Cement pastes were obtained under the same conditions as in Run 1 except that calcium hydroxides having average particle diameters of 62.0  $\mu\text{m}$ , 23.5  $\mu\text{m}$ , 6.5  $\mu\text{m}$ , 3.8  $\mu\text{m}$  and 3.3  $\mu\text{m}$  were used to measure their setting times.

### <Results>

The results of the above Runs are shown in Table B below and Fig. 1 attached.

Table B

	Average particle diameter of $\text{Ca}(\text{OH})_2$ ( $\mu\text{m}$ )	Setting time (h:m)	
		Initial setting time	Final setting time
Run 1	2.5	1:25	1:50
Run 2	1.3	1:20	1:45
Run 3	0.5	1:10	1:40
Run 4	2.7	1:25	1:55
Comparative Run 1	-	2:10	3:30
Comparative Run 2	62.0	2:00	2:45
Comparative Run 3	23.5	1:55	2:40
Comparative Run 4	6.5	1:55	2:30
Comparative Run 5	3.8	1:45	2:15
Comparative Run 6	3.3	1:40	2:10

In table B, the average particle diameter of  $\text{Ca}(\text{OH})_2$  was obtained by the following method:  
the particle size distribution of calcium hydroxide is measured by using ethanol as a dispersion medium and a laser diffraction particle size distribution measuring instrument to calculate a volume average diameter from the measurement result as average particle diameter.

The setting time of a cement paste containing the setting accelerator is measured by a method specified in JIS R 5201

"physical testing method of cement". Calcium hydroxide is added to a cement.

<Observations>

It is understood that the initial and final setting times of Runs 1, 2, 3 and 4 in which calcium hydroxide having an average particle diameter of 3  $\mu\text{m}$  or less was used are much shorter than those of Comparative Runs 2 to 6 in which calcium hydroxide having an average particle diameter of more than 3  $\mu\text{m}$  was used.

Especially, it is noted that the initial and final setting times of Comparative Runs 2, 3 and 4 in which calcium hydroxide having an average particle diameter of 6.5  $\mu\text{m}$ , 23.5  $\mu\text{m}$  and 62.0  $\mu\text{m}$  were used are almost the same as that of Comparative Run 1 in which calcium hydroxide was not used. It is understood that the initial setting time and the final setting time drastically change when the average particle size of calcium hydroxide is 3  $\mu\text{m}$ .

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Further declarant saith not.

Hiroyoshi Kato

Hiroyoshi KATO

December 2, 2008

Date